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RESEARCH MEMORANDUM

KINETIC STUDY OF MASS TRANSFER BY SODIUM HYDROXIDE IN NICKEL UNDER FREE-CONVECTION CONDITIONS

By Don R. Mosher and Robert A. Lad

Lewis Flight Propulsion Laboratory
Cleveland, Ohio

NATIONAL ADVISORY COMMITTEE
FOR AERONAUTICS
WASHINGTON
March 10, 1954

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KINETIC STUDY OF MASS TRANSFER BY SODIUM HYDROXIDE IN NICKEL UNDER FREE-CONVECTION CONDITIONS

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SUMMARY

An investigation was conducted using static capsules fabricated from "L" nickel tubing to determine the effect of temperature level, temperature gradient, and test duration on corrosion and mass transfer by molten sodium hydroxide under free-convection conditions. A base temperature range from 1000° to 1600° F with temperature differences to 500° was studied. The rate of mass transfer was found to be strongly dependent on both temperature level and gradient. The rate shows little tendency to decrease for test durations up to 200 hours, although the concentration of nickel in the melt approaches a limiting value after 100 hours.

INTRODUCTION

The general interest in molten sodium hydroxide as a high-temperature heat-transfer medium has resulted in efforts to find a suitable container material. Molten sodium hydroxide is corrosive at high temperatures and no container material has yet been found in which corrosion and mass transfer do not occur. One portion of the results of such an investigation conducted at the NACA Lewis laboratory is reported herein. It is a study of the effects on mass transfer of three important variables - temperature level, temperature gradient, and test duration. "L" nickel was chosen for the study because its corrosion by molten sodium hydroxide is limited to nearly uniform removal of metal in the high-temperature region (with no intergranular penetration) and the formation of a crystalline nickel deposit in the cooler regions.

Static capsules, each containing a weighed disk specimen and a weighed quantity of sodium hydroxide were subjected to tests of 24 hours duration at 1400°, 1500°, and 1600° F with temperature differences up to approximately 500° F. The effect of test durations to 200 hours was studied at 1000°, 1400°, and 1500° F with a temperature difference of 83° F. Specimen weight losses were correlated with the weight of material deposited in the cool zone. The results are presented as plots of specimen weight change against temperature, temperature difference, and test duration.

APPARATUS AND PROCEDURE

Test capsules. - Work on another phase of the sodium hydroxide - nickel corrosion problem at this laboratory has shown that the results of free-convection tests are extremely sensitive to variables such as pretreatment of the nickel and caustic, the quantity of molten fluid in the capsule, and the uniformity in composition of the capsule material. The capsules for this investigation were prepared in a manner which yielded results reliable to ±10 percent for a fixed temperature and temperature gradient. This is to be compared with a variability of 200 to 300 percent which results from normal care in the control of such variables.

The test specimens were 5-inch long capsules, fabricated from 5/8-inch outside diameter by 1/32-inch wall "L" nickel tubing, set up as shown in figure 1. All portions of the capsule except the vent tube were made from the same piece of nickel stock, including the weighed disk specimen which rested at the bottom. A standardized cleaning procedure was used which consisted of an acid treatment followed by distilled water and acetone rinses. Sodium hydroxide (c.p. grade, 13.0±0.1 g) was loaded into the capsule under an atmosphere of dry nitrogen. The impurity content of the sodium hydroxide is listed in table I.

The caustic was freed from water and air by evacuation and heating to 700° F in the apparatus shown in figure 2. Evacuation and heating were continued until the pressure in the system as indicated on the thermocouple gage was below 5 microns. The capsules were then cooled to room temperature and filled with helium to approximately 2 pounds per square inch gage. The vent tubes were finally crimped and welded.

The specimens were mounted, four at a time, in the specimen holder and thermocoupled as shown in figure 3. One thermocouple located at the bottom of each capsule and one at the liquid level ($2\frac{1}{2}$ in. from the bottom) were used for temperature measurement, while a third thermocouple, also at the liquid level, was used for control of the temperature gradient.

Apparatus. - The capsules were tested in the apparatus shown in figure 4. This apparatus was inserted in a vertical Globar furnace which was a right circular cylinder. The desired axial temperature differences were achieved by directing cooling air from individual annular chambers onto the upper portion of the capsules. The air supply line to each cooler was equipped with a solenoid valve actuated by a temperature controller which used a control thermocouple from the appropriate capsule.

<u>Procedure</u>. - Prior to a test, the furnace was allowed to reach equilibrium at an operating temperature which ranged from near test temperature to some 500° higher, depending on the temperature gradient desired. The air controllers were set to maintain the liquid level temperature and the capsules were lowered into the furnace. When the desired temperatures were approached, small adjustments were usually required on the various controllers (furnace or air) to bring the temperatures to the proper level.

At the conclusion of a test the capsules were stripped of thermocouples and the oxide film was removed from the exterior. The capsules were sectioned approximately 1 inch from each end with a tubing cutter and the caustic was dissolved out with water. The water solutions were analyzed for metal pickup. Weight loss of the nickel disks and the weight of the crystalline deposit in the cold zone were determined, the latter by weighing the tube before and after reaming to the original 1/2-inch dimension.

RESULTS AND DISCUSSION

The data plotted in figure 5 indicate that there is an almost linear relation between the amount of nickel lost and the amount deposited. The deviation from linearity is believed to be the result of a variation in the flow pattern as the tube dimensions change with the deposition in the cold zone. The deposit weight is much greater than the weight loss because the latter was determined from a small fraction of the hot zone area, namely, the small disk specimen. The fact that the build-up of nickel in the melt (fig. 6) was small and approached a limiting value in the region of 100 hours leads to the conclusion that there is a 1 to 1 correspondence between loss and deposit.

The mass-transfer process begins at a rapid rate which, after an initial period, falls to a fairly constant value that is maintained for times through 200 hours. This can be seen in figure 7, which presents results obtained at three base temperatures for a temperature difference of 83° F. The fact that the rate curve shows no indication of leveling off when the nickel concentration in the melt is approaching saturation (after 100 hr) indicated that a pretreatment of the caustic to saturate it with nickel would not tend to reduce mass transfer.

The experiments with temperature difference varying up to 500° confirmed the belief that the rate of mass transfer is strongly dependent on both temperature and temperature gradient. The results plotted in figure 8 indicate a nonlinear dependence of weight gain on temperature gradient. A possible contributing factor to the decrease at the high gradients may be a convective rate too large to permit the complete saturation of the hotter fluid with nickel compounds. Another factor is that at long times and high gradients, the bore at the liquid level becomes choked with deposit and the zone of deposition moves downward into regions of correspondingly smaller temperature gradient.

The nickel concentrations at 200 hours obtained from figure 6 were used to make a plot of nickel concentration against temperature. The curve so obtained is shown in figure 9. The curve is characteristic of materials for which heat is absorbed on solution.

SUMMARY OF RESULTS

The result of an investigation of mass transfer in the sodium hydroxide - nickel system under free-convection conditions in the temperature range 1000° to 1600° F can be summarized as follows:

1. Specimen weight loss in the hot zone had an almost linear relation to the weight of nickel deposited in the cool zone.

2. The nickel concentration in the melt approached a limiting value after 100 hours.

3. The rate of mass transfer was very sensitive to both base temperature and temperature gradient.

4. The mass-transfer rate reached a constant value in about 30 hours. No further decrease in rate was observed for times as long as 200 hours.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, December 3, 1953

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TABLE I. - ANALYSIS OF SODIUM HYDROXIDE PELLETS[a]

Material	Percent by weight
Assay (NaOH)	97.6
Chloride (Cl)	.005
Iron (Fe)	.001
Other heavy metals (as Ag)	.000
Carbonate (Na_2CO_3)	.32
Phosphate (PO_4)	.000
Silica and NH_4OH precipitate	.000
Total nitrogen (as NH_3, NO_2)	.001
Sulfate (SO_4)	.000

[a]As per label and verified by analysis at the time of use.

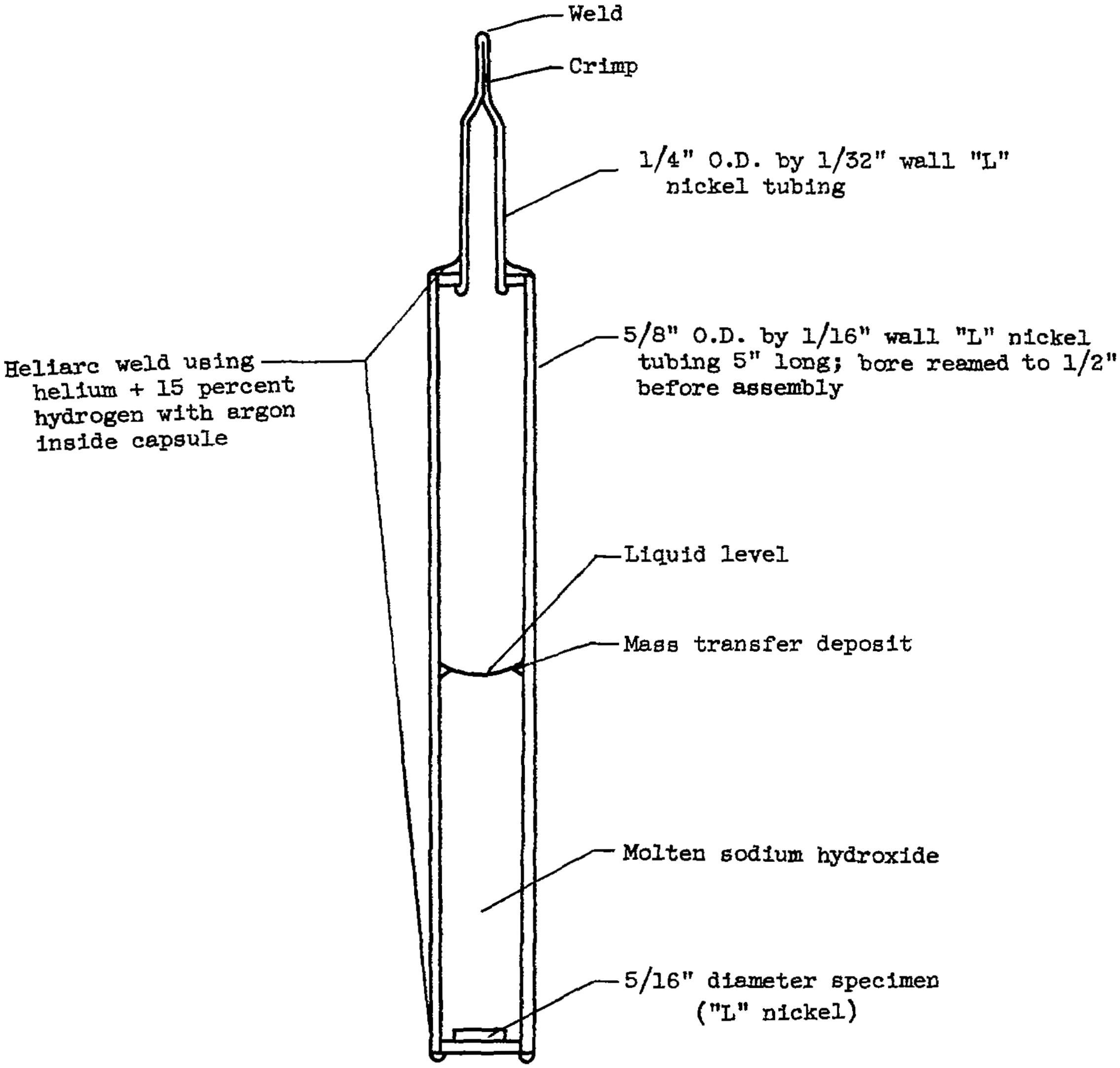

Figure 1. - Test Capsule

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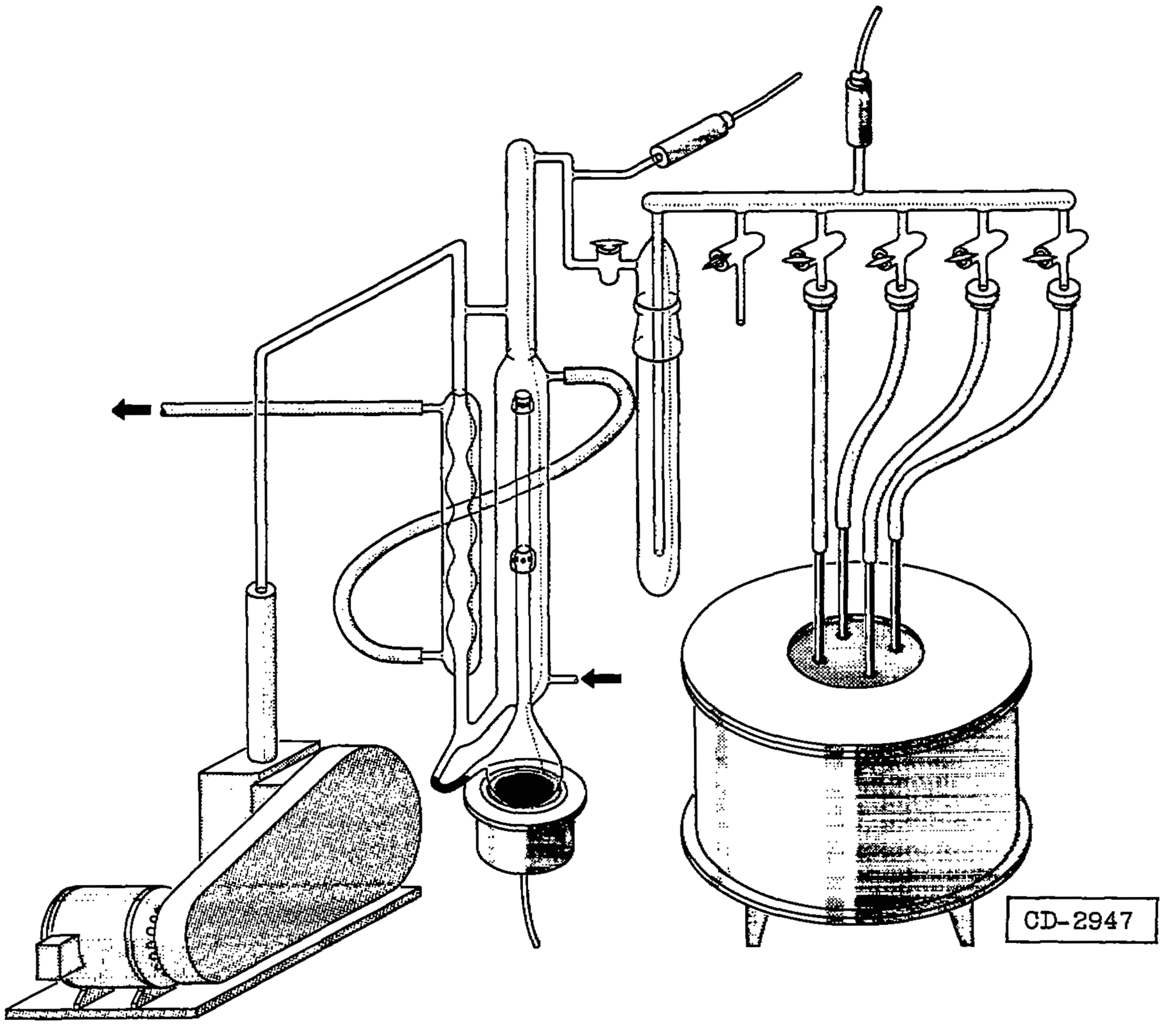

Figure 2. - Apparatus for dehydration of caustic.

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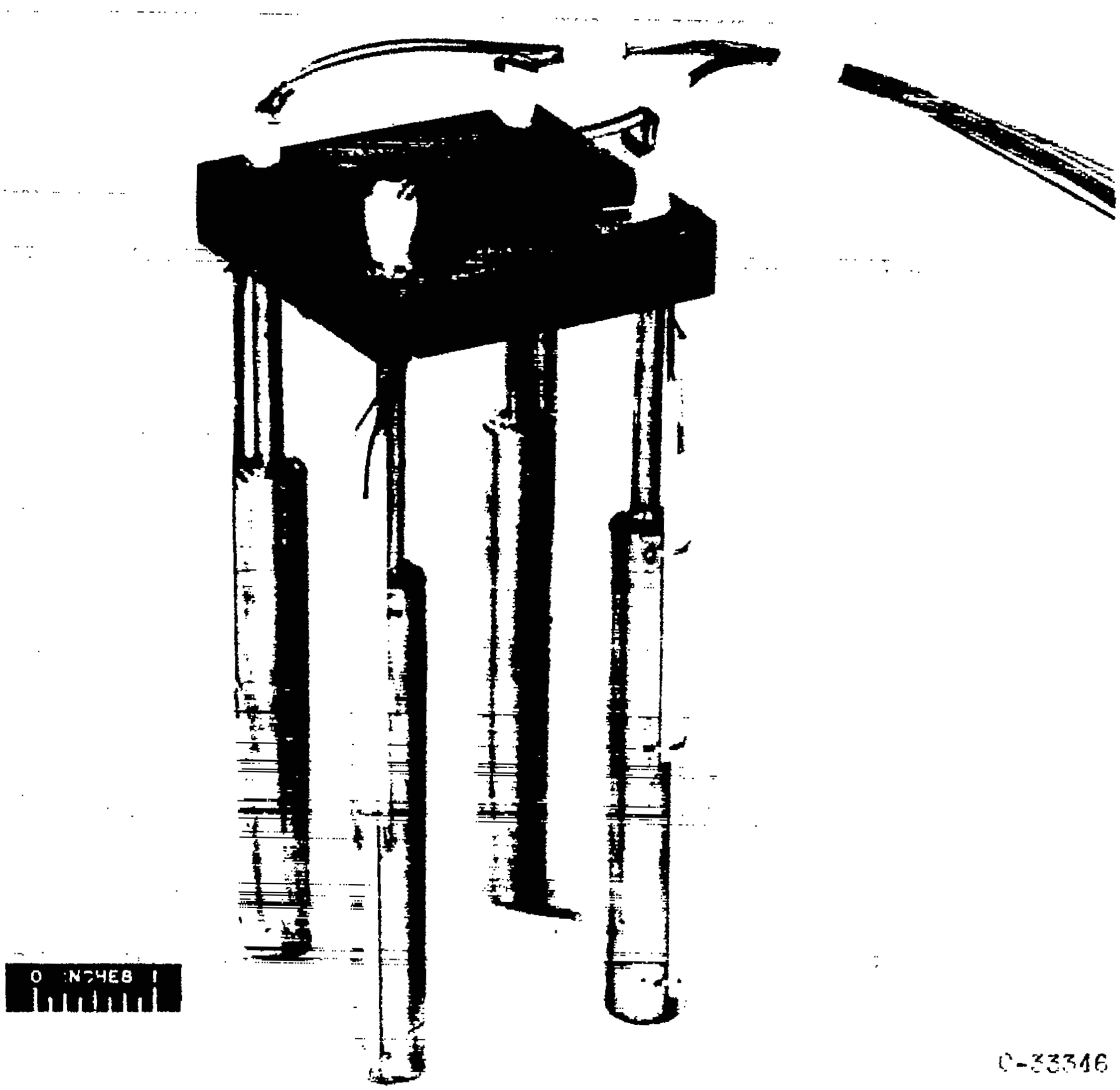

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Figure 3. - Instrumented specimens.

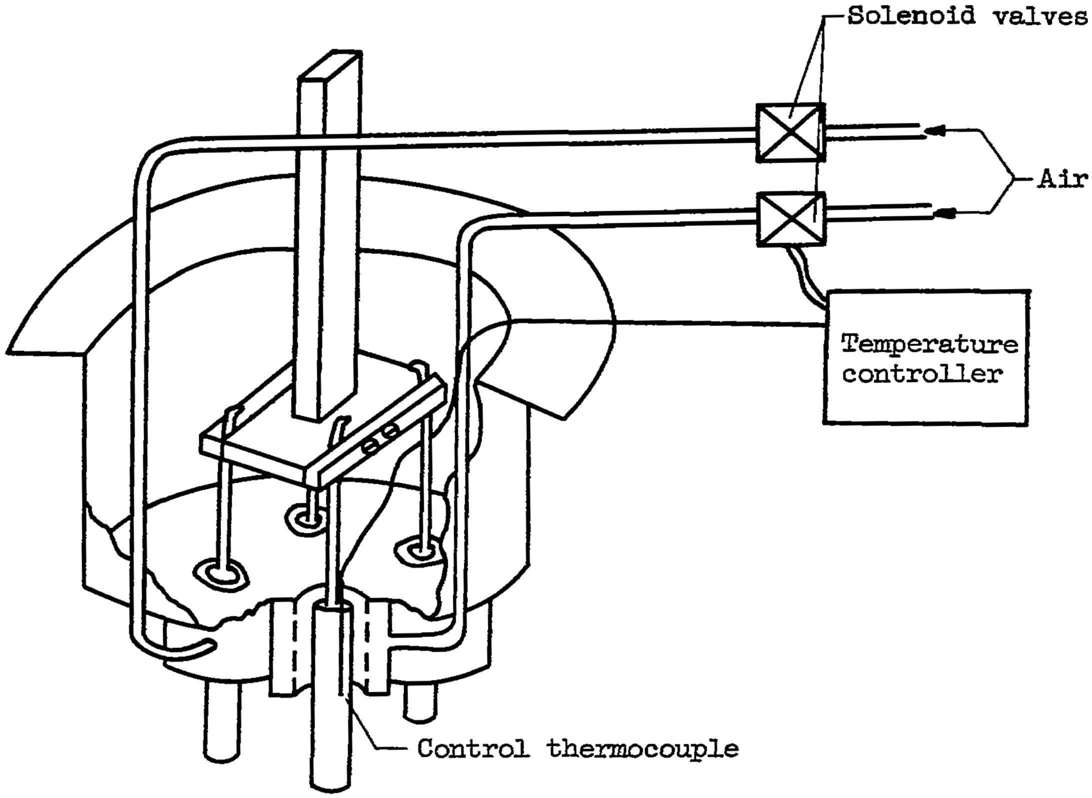

Figure 4. - Apparatus for controlling temperature gradients.

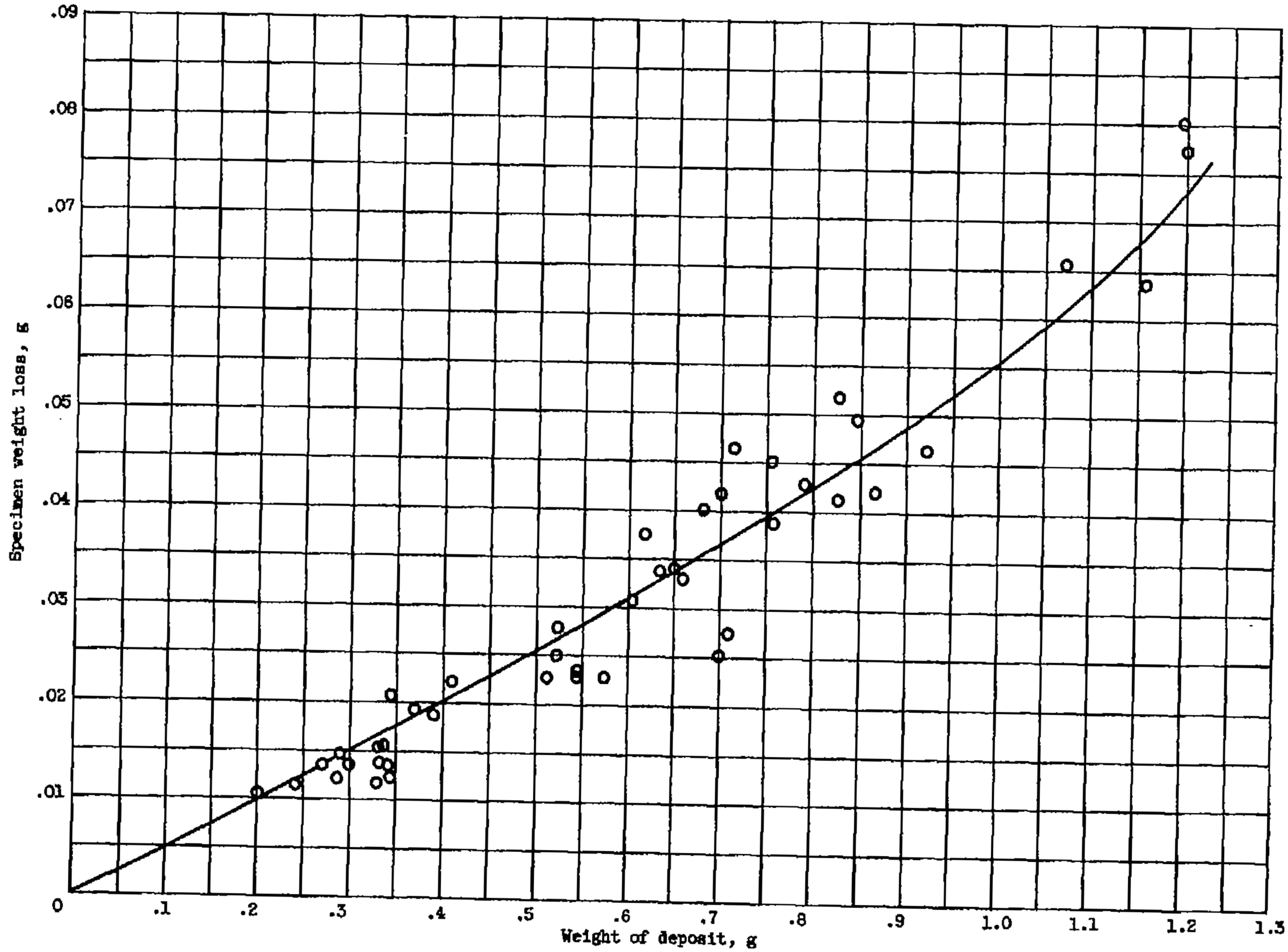

Figure 5. - Specimen weight loss against weight of deposit for wide variety of temperatures, temperature gradients, and test durations.

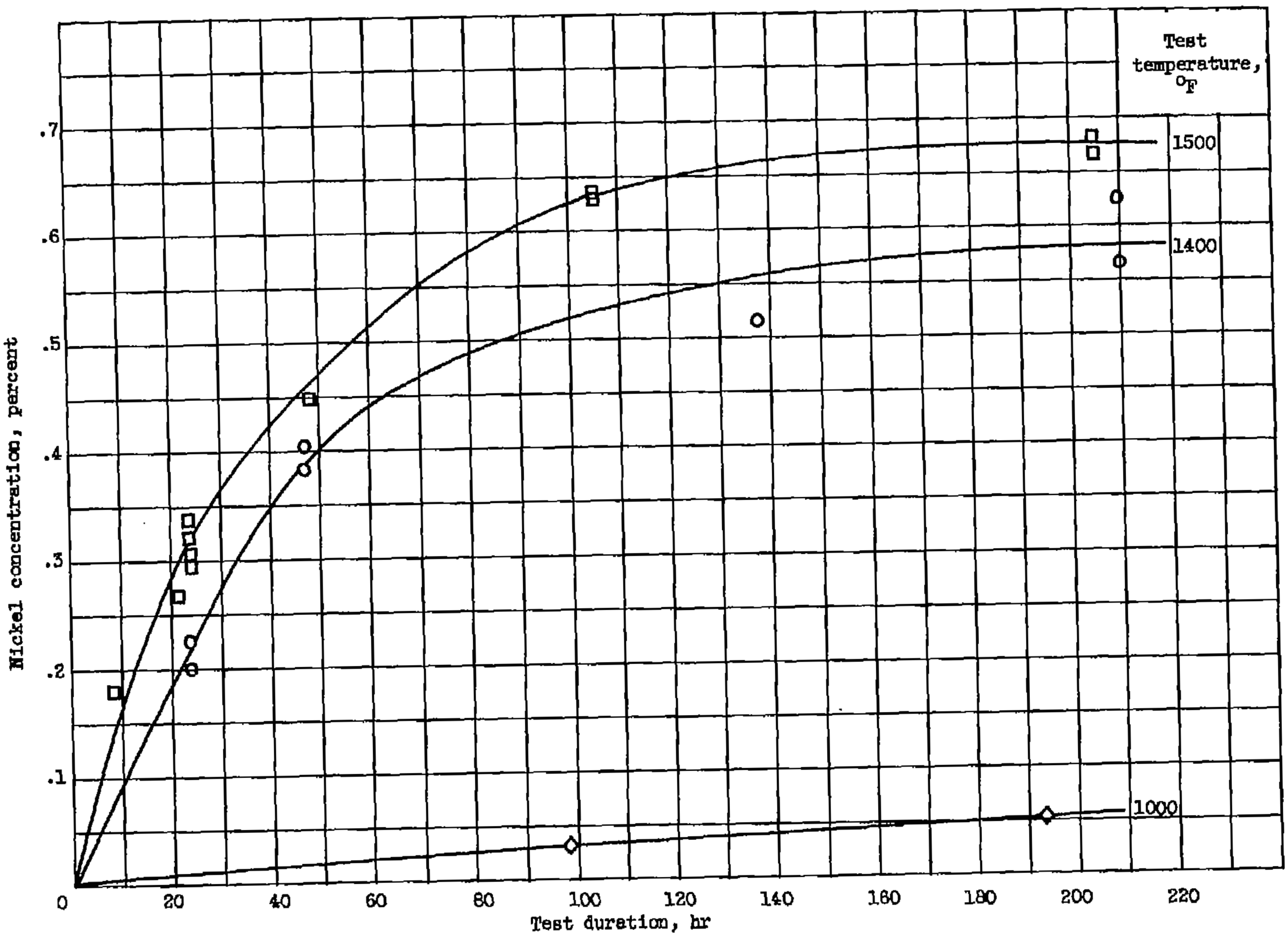

Figure 6. - Concentration of nickel in sodium hydroxide against test duration for temperature difference of 83°±15° F.

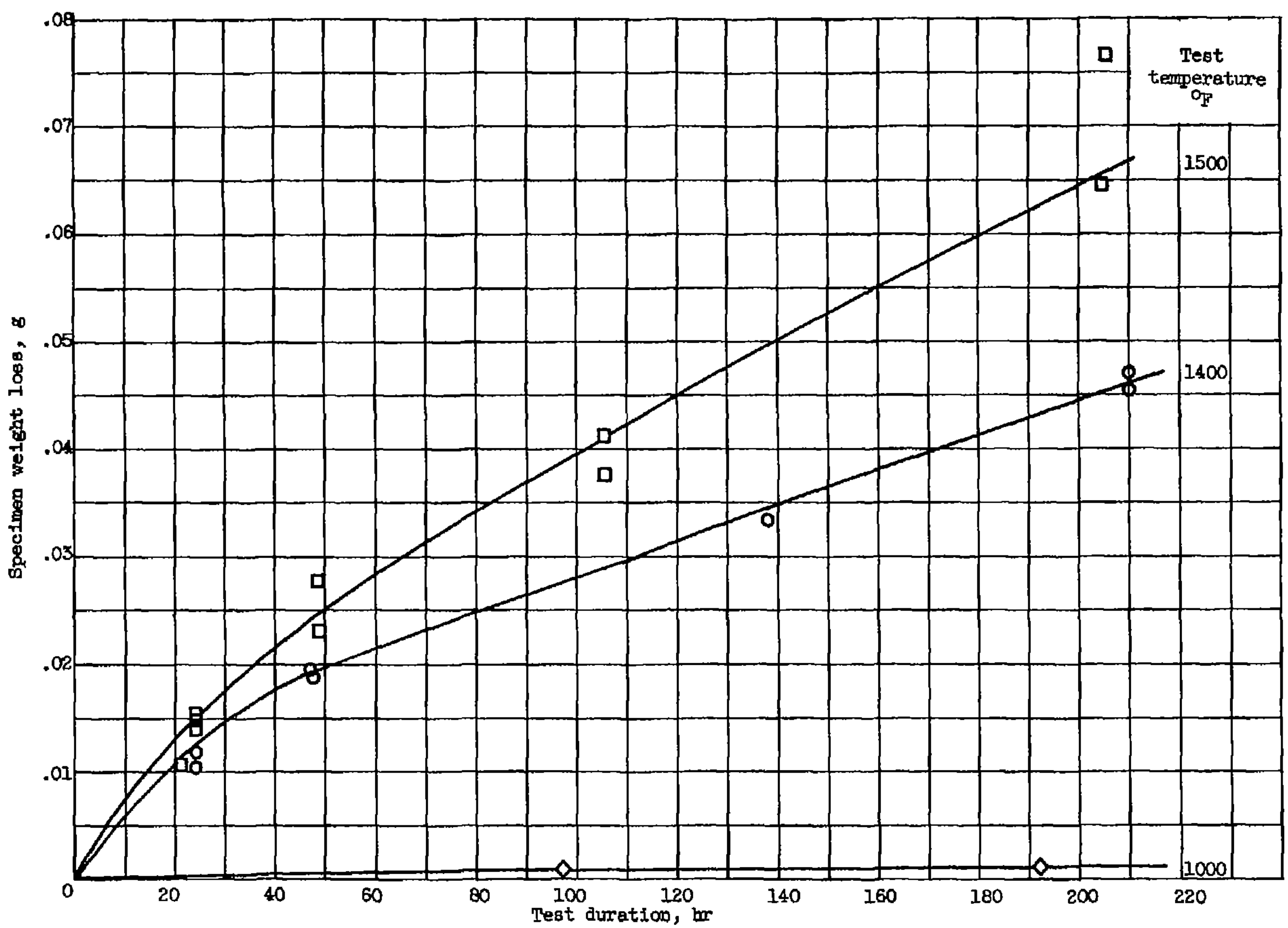

Figure 7. - Specimen weight loss against test duration for temperature difference of $83^{\circ}\pm15^{\circ}$ F.

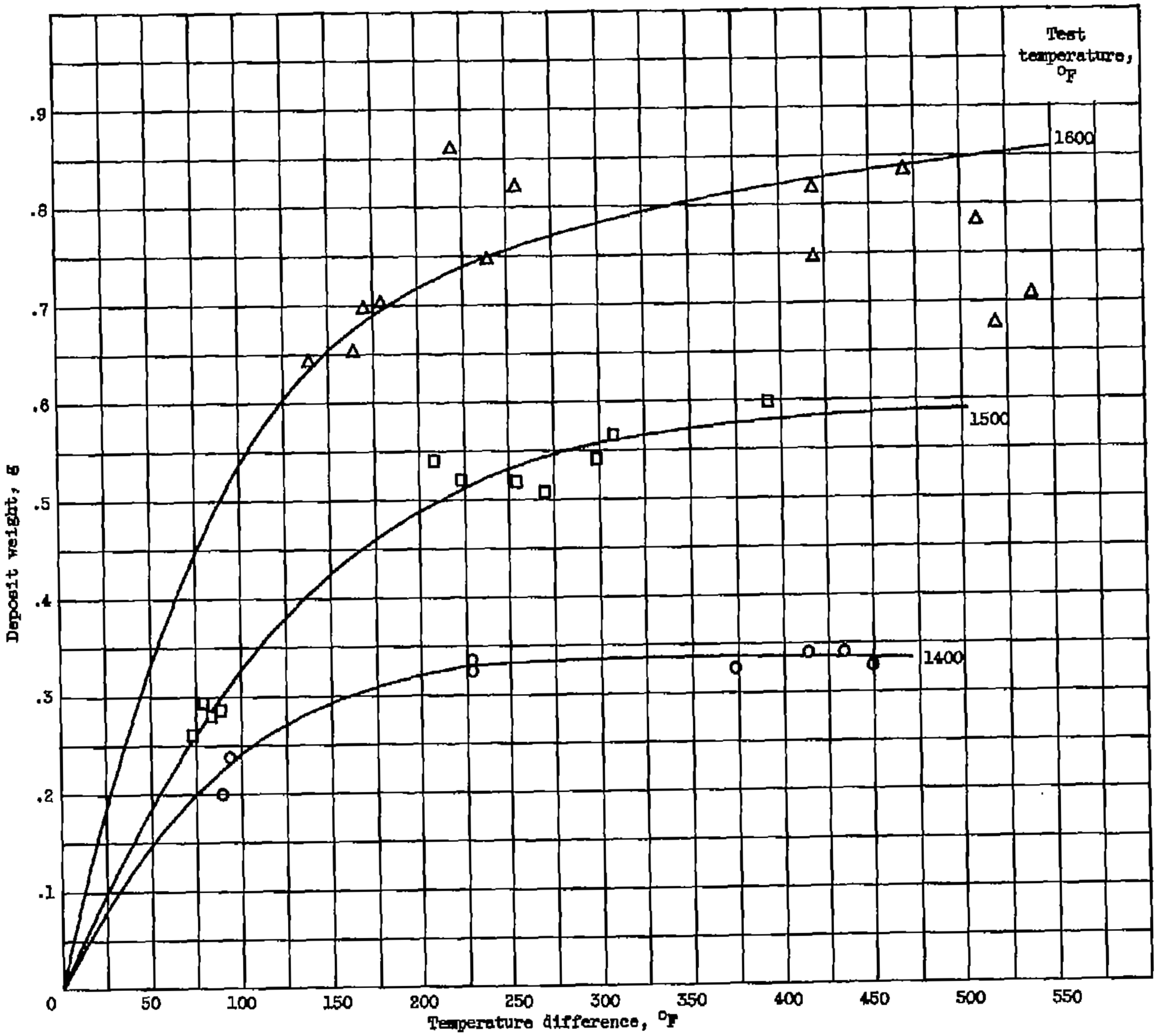

Figure 8. - Deposit weight as function of temperature difference for 24-hour tests.

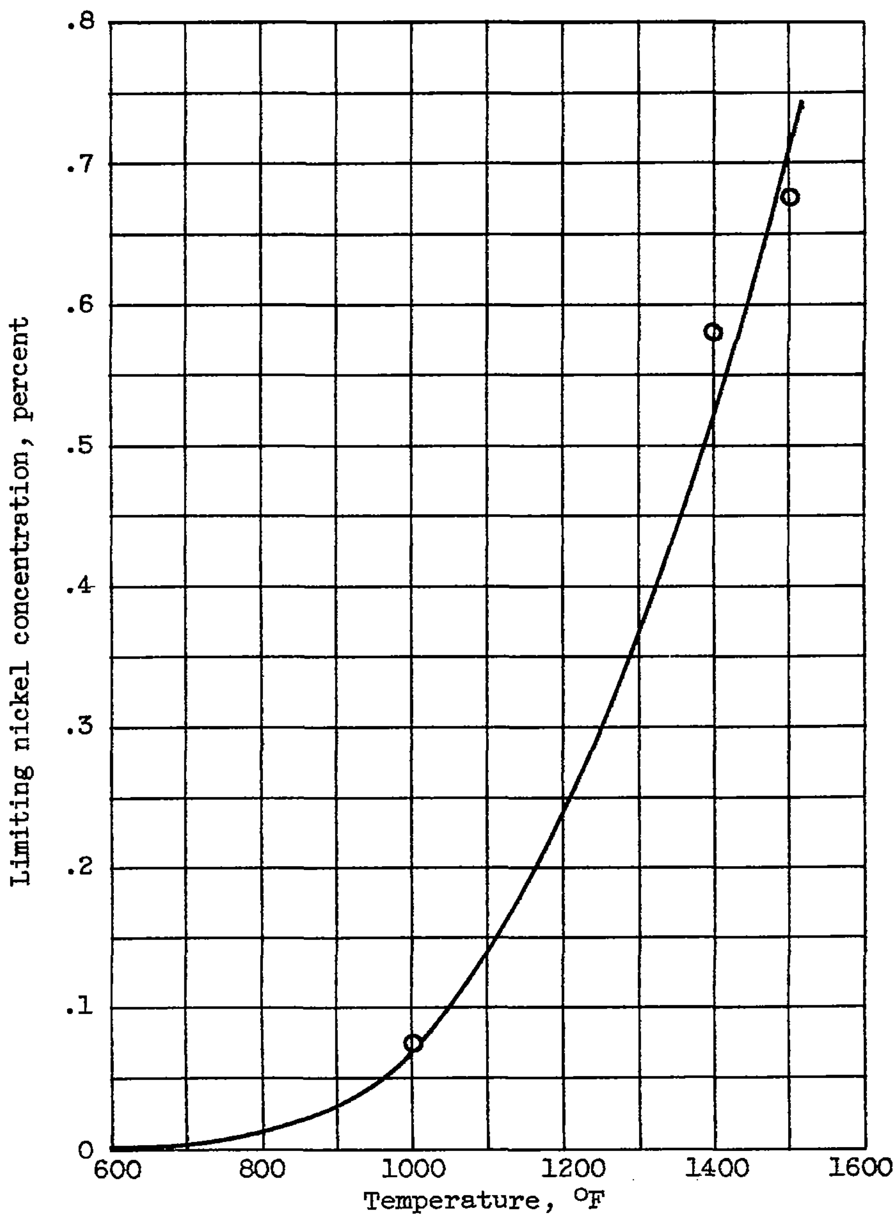

Figure 9. - Nickel concentration in sodium hydroxide after 200 hours as function of test temperature.

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